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IS 9343 (1979): 1-phenyl-3-pyrazolidone, photographic grade [CHD 5: Electroplating Chemicals and Photographic Materials]

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IS : 9343 - 1979

Indian Standard

SPECIFICATION FOR 1-PHENYL-3-PYRAZOLIDONE, PHOTOGRAPHIC GRADE

UDC 547.775 : 771.7

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

***June* 1980**

AMEDDMENT NO. 1 AUGUST 1986

TO

IS: 9343-1979 SPECIFICATION FOR
1-PHENYL-3-PYRAZOLIDONE, PHOTOGRAPHIC GRADE

(*Page 11, clause B-9.1*) - Add the following at the end:

'The colour of the solution shall not be more than light orange.'

(CDC 44) Reprography Unit , ISI, New Delhi, India

Indian Standard

SPECIFICATION FOR 1-PHENYL-3-PYRAZOLIDONE, PHOTOGRAPHIC GRADE

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(*Continued on page* 2)

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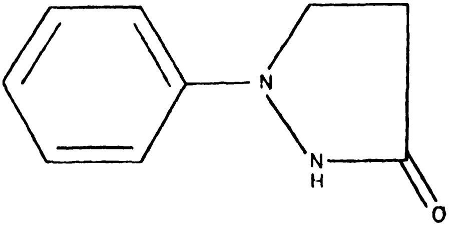
Indian Standard

SPECIFICATION FOR 1-PHENYL-3-PYRAZOLIDONE, PHOTOGRAPHIC GRADE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 27 November 1979, after the draft finalized by the Photographic Chemicals and Related Materials Sectional Committee had been approved by the Chemical Division Council.

0.2 1-Phenyl-3-pyrazolidone also called 1-phenyl-3-pyrazolidinone, is used as a photographic developer. It is known in the trade as ' phenidone '. It has the structural formula:



MOLECULAR FORMULA $C_9H_{10}N_2O$;
MOLECULAR MASS 162

0.3 In the preparation of this standard, assistance has been derived from ISO 3299-1976 Photographic grade 1-phenyl-3-pyrazolidinone (phenidone) — Specification, prepared by the International Organization for Standardization (ISO).

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

---

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for l-phenyl-3-pyrozolidone used for processing photographic films and papers.

2. REQUIREMENTS

2.1 Description The material shall be in the form of white to pale cream crystalline powder, free from aggregates or large crystals.

2.2 The material shall pass the identity tests proscribed in Appendix A.

2.3 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods prescribed in Appendix B. Reference to the relevant clauses of Appendix B is given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR 1-PHENYL-3-PYRAZOLIDONE

SL. No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL. No. IN APPENDIX B)
(1)	(2)	(3)	(4)
i)	Assay, percent by mass, *Min*	98.5	B-2
ii)	Ash, percent by mass, *Max*	0.1	B-3
iii)	Heavy metals (as Pb), percent by mass. *Max*	0.001	B-4
iv)	Iron (as Fe), percent by mass, *Max*	0.005	B-5
v)	Volatile matter at 65°C, percent by mass, *Max*	0.1	B-6
vi)	Solubility in alkaline sulphite solution	To pass the test	B-7
Vii)	Matter insoluble in chloroform, percent by mass, *Max*	0.1	B-8
viii)	Solubility in water	To pass the test	B-9

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in suitable dark coloured air-tight bottles or as agreed to between the purchaser and the supplier.

3.2 Marking — The containers shall be securely closed and legibly and indelibly marked with the name of the manufacturer, and recognized trade-mark, if any; the date of the manufacture; mass of the material in the containers; and batch number in code or otherwise to enable the lot of manufacture to be traced from records.

3.2-1 The containers may also bo marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn and their conformity to the standard determined as prescribed in Appendix C.

APPENDIX A

(*Clause* 2.2)

DENSITY TESTS FOR 1-PHENYL-3.PYRAZOLIDONE

A-1. PHYSICAL APPEARANCE

A-1.1 Procedure — Examine the material on a watch-glass under direct day light. Observe for conformity of the material to the requirements laid down in **2.1.**

A.2. MELTING POINT

A-2.1 Procedure — Assemble capillary tube melting point apparatus using a thermometer which covers the range 0 to 200°C. Prepare three capillary tubes placing in them (a) sample to be tested, (b) a sample of known melting point, and (c) finely ground mixtures of (a) and (b) mixed in the ratio 1 : 1. Identify the tubes and attach them to the thermometer. Heat the bath to approximately 100°C and thereafter raise the temperature at the rate of 1°C per minute, stirring constantly. Note the melting point of each as indicated by the first appearance of liquefaction. The melting point of the mixture shall be not lower than that of the two samples. The test sample shall melt in between 120 to 123°C.

APPENDIX B

(*Clause* 2.3, and *Table* I)

METHODS OF TEST FOR 1.PHENYL-3-PYRAZOLIDONE

B-1. QUALITY OF REAGENTS

B-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

B.2. DETERMINATION OF 1.PHENYL.3.PYRAZOLIDONE CONTENT (ASSAY)

B-2.0 General — Iodometric method is used for the assay.

B-2.1 Reagents

B-2.1.1 ***Methanol***

B-2.1.2 *Buffer Solution* (p*H*4) — Dissolve 96 g of glacial acetic acid and 54.5 g of sodium acetate trihydrate in water and dilute to 1 000 ml.

B-2.1.3 *Iodine Solution* — 0.05 N solution containing 60 g of potassium iodide per litre.

B-2.1.4 *Standard Sodium Thiosulphate Solution* — 0.05 N.

B-2.1.5 *Starch Indicator* — 0.05 percent aqueous solution, freshly prepared.

B-2.2 Procedure — Weigh accurately 0.180 ± 0.005 g of the material and dissolve it in 10 ml of methanol. Dilute this solution with 75 ml of boiled water which has been cooled to between 20 to 25°C and add 25 ml of the buffer solution. Add 50 ml of the iodine solution from a burette, immediately add starch indicator and titrate, using standard sodium thiosulphate solution within 15 seconds of the addition of the iodine solution. Carry out a blank simultaneously.

B-2.3 Calculation

Assay, expressed as phenyl pyrazolidone ($C_9H_{10}N_2O$), percent by mass $= \frac{8\cdot 1 (50 N_1 - N_2 V_2)}{M}$

---

*Specification for water for general laboratory use (*second revision*).

where

N_1 = normality of the iodine solution,

N_2 = normality of the sodium thiosulphate solution,

V_2 = volume in ml of sodium thiosulphate solution required for back titration, and

M = mass in g of the sample taken.

B-3. DETERMINATION OF ASH

B-3.1 Procedure — Incinerate about 5 g of the material, accurately weighed, in a tared silica crucible after moistening with a few drops of concentrated sulphuric acid and then ignite at dull red heat (approximately 600°C). Cool in a desiccator and weigh till constant mass is obtained. Preserve the residue for the heavy metals and iron tests.

B-3.2 Calculation

$$\text{Ash, percent by mass} = \frac{M}{M_1} \times 100$$

where

M = mass in g of the residue, and

M_1 = mass in g of the material taken for the test.

B-4. DETERMINATION OF HEAVY METALS

B-4.0 General — The colour produced by heavy metals in the sample on treating with hydrogen sulphide is compared with that produced with standard lead solution under identical conditions.

B-4.1 Reagents

B-4.1.1 *Sodium Carbonate Anhydrous*

B-4.1.2 *Dilute Hydrochloric Acid* — approximately 10 percent and 1 percent (*v/v*).

B-4.1.3 p-*Nitrophenol Indicator* — 0.25 percent aqueous solution.

B-4.1.4 *Ammonium Hydroxide* — 10 percent.

B-4.1.5 *Hydrogen Sulphide* — saturated aqueous solution.

B-4.1.6 *Standard Lead Solution* — Dissolve 0.64 g of lead nitrate in water, add 10 ml of nitric acid (relative density 1.42) and dilute to 1 000 ml with water. Further dilute 10 ml to 1 000 ml water when required. One millilitrc of the resulting solution will contain 4 micrograms of lead.

B-4.2 Procedure

B-4.2.1 Fuse the residue obtained from the determination of ash (*see* **B-3.1**) with 1 g of sodium corbonate, anhydrous, Cool and dissolve the residue in 15 nd of dilute hydrochloric acid (10 percent) and warm on a steam bath for 10 minutes. Cool, dilute to 50 ml with water in a volumetric flask and mix thoroughly. Use 20 ml of this solution for heavy metals test and reserve the remaining solution for the determination of iron.

B-4.2.2 To the 20-ml portion of the sample solution in a Nessler cylinder add a few drops of *p*-nitrophenol indicator and then add ammonium hydroxide, dropwise, until the solution turns yellow. Add dilute hydrochloric acid (1 percent), dropwise, until the solution becomes colourless again and then add 2.5 ml in excess. Add 5 ml of the hydrogen sulphide solution, dilute to 50 nd and mix.

B-4.2.3 At the same time transfer 5 ml of standard lead solution into another Nessler cylinder, add 0.5 g of sodium carbonate, anhydrous and 6 ml of dilute hydrochloric acid (10 percent) and render it neutral to *p*-nitrophenol indicator by using ammoniun hydroxide and dilute hydrochloric acid as described in **B-4.2.2.** Add 2.5 ml of dilute hydrochloric acid in excess and 5 ml of hydrogen sulphide solution. Dilute to 50 ml and mix.

B-4.2.4 The material shall be considered to have not exceeded the limits of heavy metals if the intensity of colour produced in the sample solution is not more than that produced in the standard solution.

B-S. DETERMINATION OF IRON

B-5.0 General — Two methods have been prescribed, namely, (a) *o*-phenanthroline method, and (b) thiogylcollic acid method. In case of dispute, the iron content shall be determined by *o*-phenanthroline method.

B-5.1 *o*-Phenanthroline Method

B-5.1.0 *General* — *o*-phenanthroline, hydroxylamine hydrochloride solution and acetate buffer solution of *p*H 5 in equal quantities arc mixed with the material and the colour is compared with that produced by standard iron solution under identical conditions.

B-5.1.1 ***Reagents***

B-5.1.1.1 *Acetate buffer* — *p*H 5. Add 23 g of anhydrous sodium acetate to 58 ml of 2 molar acetic acid and dilute to 1 litre with water. Adjust the final *p*H of the solution to 5.0 + 0.1 with acetic acid or 10 percent sodium hydroxide solution.

B-5.1.1.2 *Hydroxylnminc hydrochloride* 10 percent aqueous solution.

B-5.1.1.3 *o-phenanthroline* (*1 : 10 phenanthroline*) — 0.1 percent aqueous solution.

B-5.1.1.4 *Standard iron solution* — Dissolve 7.02 g of crystalline ferrous ammonium sulphate [$Fe(NH_4)_2(SO_4).6H_2O$] in about 300 ml of water containing 2 ml of sulphuric acid (relative density 1.84) and dilute to 1 000 ml with water. Further dilute 5 ml to 1 000 ml in volumetric flask. One millilitre of the diluted solution contains 5 micrograms of iron (as Fe).

B-5.1.2 *Procedure* — Thoroughly mix equal parts of *o*-phenanthroline, hydroxylamine hydrochloride and acetate buffer of *p*H 5. Add 5 mi of this mixture into 20 ml portions of standard iron solution (*sec* **B-5.1.1.4**) and the sample solution (*see* **B-4.2.1**). Allow to stand for 10 minutes. Dilute to 50 ml with water and mix well.

B-5.1.3 The material shall be taken to have not exceeded the limit prescribed for iron if the intensity of colour produced is not deeper than that produced by the standard iron solution.

B-5.2 Thioglycollic Acid Method

B-5.2.1 *Reagents*

B-5.2.1.1 *Dilute hydrochloric acid* — 10 percent.

B-5.2.1.2 *Thioglycollic acid* — 89 percent purity, minimum.

B-5.2.1.3 *Ammonia Solution* — Relative density 0.90.

B-5.2.1.4 *Cilric acid solution* 20 percent (*m/v*) aqueous solution.

B-5.2.1.5 *Standard iron solution* — Dissolve 7.02 g of crystalline ferrous ammonium sulphate [$Fe(NH_4)_2(SO_4).6H_2O$] in about 300 ml of water containing 2 ml of sulphuric acid (relative density 1.84) and dilute to 1 000 ml with water. Further dilute 20 ml to 1 000 ml in volumetric flask. One millilitre of the resulting solution is equivalent to 20 microgram of iron (as Fe).

B-5.2.2 *Procedure*

B-5.2.2.1 Dissolve the residue from **B-3.1** in 1 ml of concentrated hydrochloric acid and a little water by gently warming and make up the solution to 50 ml in a volumetric flask. Transfer 20 ml aliquot of this solution to a Nessler cylinder, make just alkaline with ammonium hydroxide and dilute to 50 ml. Into a second Nessler cylinder containing 6 ml of dilute hydrochloric acid, pipette 5.0 ml of standard iron solution and dilute to about 50 ml with water. To both cylinders add 20 ml of citric acid solution and 2 drops of thioglycollic acid. Mix, make just alkaline with ammonia solution and dilute to 100 ml.

B-5.2.2.2 The material shall be taken to have not exceeded the limit prescribed for iron if the intensity of colour produced is not deeper than that produced by the standard iron solution.

B-6. DETERMINATION OF VOLATILE MATTER AT 65°C

B-6.1 Procedure — Weigh to the nearest 0.1 g, a portion of about 5 g of the sample into a previously weighed squat-form, glass stoppered weighing bottle. Dry at 65°C for 4 hours. Cool in a desiccator and weigh to the nearest 1 mg.

B-6.2 Calculation

$$\text{Volatiie matter at 65°C, percent by mass} = \frac{M_2 - M_3}{M_2 - M_1} \times 100$$

where

M_1 = mass in g of the weighing bottle,

M_3 = mass in g of the bottle and test portion before drying, and

M_3 = mass in g of the bottle and test portion after drying.

B.7. TEST FOR SOLUBILITY IN ALKALINE SULPHITE SOLUTION

B-7.1 Procedure — Dissolve 0.5 g of the sample in 1 000 ml of an aqueous solution of 25 g of sodium sulphite, anhydrous, and 25 g of sodium carbonate anthydrous. Examine the solution for clarity and colour.

B-7.2 The solution shall be considered to have passed the test if it is clear. A pink colouration, however, is permissible.

B-8. DETERMINATION OF MATTER INSOLUBLE IN CHLOROFORM

B-8.1 Procedure — Weigh to the nearest 0.1 g about 5 g of the sample and dissolve in 100 ml of chloroform. If any undissolved material remains, filter through a weighed sintered glass crucible No. G 3 and wash with 25 ml of chloroform. Dry at 105°C for 4 hours. Cool in a desiccator and weigh till constant mass is obtained.

B-8.2 Calculation

$$\text{Matter insoluble in chloroform, percent} = \frac{M_1 - M_2}{M} \times 100$$

where

M_1 = mass in g of the curcible and the residue,

M_2 = mass in g of the crucible, and

M = mass in g of the material taken for the test.

B-9. TEST FOR SOLUBILITY IN WATER

B-9.1 Procedure — Dissolve 2 g of material in 250 ml of hot (60°C) water. The material shall be considered to have passed the test if the solution is dear and free from visible suspended matter.

APPENDIX C

(*Clause* 4.1)

SAMPLING OF 1-PHENYL-3-PYRAZOLIDONE

C-1. GENERAL REQUIREMENTS FOR SAMPLING

C-1.1 In drawing, preparing, storing and handling test samples, the following precautions shall be observed.

C-1.2 Samples shall not be taken at a place exposed to weather.

C-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

C-1.4 To draw representative samples, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

C-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass or plastic containers.

C-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

C-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the year of manufacture.

C-1.8 Samples shall he stored in such a manner that the temperature of the material does not vary unduly from ambient temperature.

C-2. SCALE OF SAMPLING

C-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches, the batches shall be marked separately and the group of containers in each batch shall constitute separate lot.

C-2.2 From each lot five containers shall be selected (*see* **C-2.2.1**) for drawing samples for test. In case the lot contains less than five containers, each container shall be tested.

C-2.2.1 These containers shall be selected at random from the lot. In order to ensure the randomness of selection, procedure given in IS : 4905-1968* may be followed. In case this standard is not available the following procedure may be adopted:

> ' Arrange all the containers in the lot in a systematic manner and starting from any one, count them as 1, 2,......................., *r*, where r is the integral part of *N/n* (*N* and *n* being the lot size and sample size respectively). Every *r*th container thus counted shall be withdrawn to constitute the sample.'

C-3. PREPARATION OF TEST SAMPLES

C-3.1 Drawing of Samples — Draw a representative sample from each container selected.

C-3.1.1 Mix portions of the material drawn from the same container to prepare the test sample representing the container. The quantity of material to be drawn from each container shall be suflicient to conduct all the tests given in 2 and shall be not less than 200 g.

C-3.1.2 The samples shall be stored in dry glass bottles or other suitable containers, sealed air-tight and labelled with all the particulars of sampling given in **C-1.7.**

C-3.L3 Five test samples representing the five containers selected as in **C-2.2** shall constitute a set of test samples.

---

*Methods for random sampling.

C-3.2 Out of the portions of material from all the five containers selected, approximately equal quantity of the material shall be taken and mixed together to form a composite sample of quantity sufficient to carry out triplicate determinations for each of the characteristics tested on the composite sample.

C-3.2.1 The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and third to be used as a referee sample.

C-3.3 The remaining portion of the material from each of the five containers shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the container samples shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

C-3.4 All the individual and composile samples shall be immediately transferred to separate sample containers and labelled with full identification particulars.

C-3.5 The referee samples constituting of a composite sample and a set of individual samples shall bear the seal of both the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of dispute between the two.

C-4. NUMBER OF TESTS

C-4.1 Tests for the determination of 1-phenyl-3-pyrazolidone, heavy metals and iron shall be conducted on each of the five individual samples. Tests for all other characteristics prescribed in 2 and Table 1 shall be conducted on the composite sample.

C-5. CRITERIA FOR CONFORMITY

C-5.1 Tests Carried out on Five Individual Test Samples

C-5.1.1 *1-Phenyl-3-parazolidone* — From the test results for 1-phenyl-3-pyrazolidone, the mean ($\bar{X}$) and range (R) of test results shall be calculated (range being defined as the difference between the maximum and the minimum values of test results). The lot shall be considered to have passed in respect of assay if the value of the expression ($\bar{X} \pm 0.6\ R$) is greater than or equal to 98.5.

C-5.1.2 *Heavy Metals and Iron* — The lot shall be considered to have passed for the characteristics tested if each test sample passes the corresponding test prescribed.

C-5.2 Test Carried out on Composite Test Sample — The lot shall be considered to have passed in respect of the characteristics tested on the composite test sample (*see* **C-4.1**) if the composite tost sample satisfies the corresponding tests prescribed.

C-5.3 The lot shall be considered as conforming to the specification if it satisfies all the criteria given in **C-5.1** and **C-5.2.**

INDIAN STANDARDS
ON
PHOTOGRAPHIC CHEMICALS

IS:

246-l972	Sodium thiosnlphate, crystalline (*third revision*)
247-1972	Sodium sulphire, anhvdrons (*third revision*)
248-1978	Sodium bisnlphite (sodium metabisulphite) (*third revision*)
332-1967	Chromium potassium sulphate (chrome alum) (*first revison*)
388-1972	Hydroquinone, photographic grade (*second revision*)
500-1972	Potassium metabisulphite, photographic grade (*second revision*)
557-1968	Sodium acetate, technical and photographic (*first revision*)
2211-1972	Anhydrous sodium thiosulphate, photographic 'grade (*first revision*)
2318-1974	Silver nitrate, photographic grade (*first revision*)
2797-1964	Potassium bromide
4l73-1975	4-Methylammophenol sulphate (*first revision*)
5379-1969	Ammonium thiosulphate, photographic grade
5380-1976	Sodium bromide, photographic grade (*first revision*)
5381-1969	Quantity packaging of sensitized photographic materials
5431-1969	Definition of motion-preture safety films
6139-1971	Sizes of photographic paper for general use
6212-1971	Method for the determination of residual thiosulphate in processed black and white photographic films and plates
6650-1972	Method for determination of residual thiosulphate and telrathionate in processed photographic papers
6678-1972	Ammonium thiosulphate solution, photographic grade
8281-1976	Hydroxylamine sulphate, photographic grade

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Gangotri Complex, Bhadbhada Road, T. T. Nagar	BHOPAL 462003	6 27 16
22E Kalpana Area	BHUBANESHWAR 751014	5 36 27
Ahimsa Bldg, SCO 82-83, Sector 17C	CHANDIGARH 160017	2 83 20
5-8-56C L. N. Gupta Marg	HYDERABAD 500001	22 10 83
D-277 Todarmal Marg, Banipark	JAIPUR 302006	6 98 32
117/418 B Sarvodaya Nagar	KANPUR 208005	8 12 72
Patliputra Industrial Estate	PATNA 800013	6 28 08
Hantex Bldg (2nd Floor), Rly Station Road	TRIVANDRUM 695001	32 27

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